

ARMY RESEARCH LABORATORY



Recovery of Shock-Loaded XM46 Liquid Propellant

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13. ABSTRACT (Maximum 200 words) <p>The response of XM46 liquid propellant (LP) to a single shock wave is studied. LP commonly experiences shock-release-recompression when impacted in a storage container. The goal here is to determine whether during such an event potentially sensitizing chemical reactions occur during passage of the initial shock wave. Shocked LP samples were recovered and analyzed for evidence of chemical reaction. The technique for recovering shocked LP samples was developed by using water as an inert surrogate for liquid propellant. Recovery was successful after a shock of 4.8 GPa. Seven additional tests were conducted on XM46; some additional technique modification was required during these tests. Both the development of the recovery technique and the chemical response of the LP are discussed. For a shock in the range of 5.1-6.1 GPa, the LP burned. This somewhat contradicts previous LP sensitivity results; several possible explanations are given. For additional tests in the range of 1.1-3.6 GPa, no visible evidence of reaction was observed. Fourier transform infrared/cylindrical internal reflectance (FTIR/CIR) and microreflectance FTIR spectroscopy of recovered samples generally supported this observation. From these limited data it is tentatively concluded that no significant chemical reactions occur during shock of virgin LP at less than 2.4 GPa. More tests are recommended to confirm this conclusion.</p>				
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1. INTRODUCTION

The U.S. Army Research Laboratory (ARL) was funded by PM-Crusader to recover shocked XM46 liquid propellant (LP) and search for evidence of chemical reaction products. Vulnerability testing on LP at ARL has demonstrated that XM46 is extremely insensitive to a single shock wave, but it is markedly more sensitive to a second compression following release of the incident shock. During recompression, ignition most likely occurs through compression of gaseous region within the LP. Thus, the goal here was to identify whether sensitization occurred during the initial shock, by some chemical reaction creating dissolved gases, or during shock release, by cavitation creating voids. The approach was to expose a small quantity of XM46 liquid propellant to a single shock wave, recover the sample, and search for evidence of chemical reactions which may sensitize the LP to recompression. This work was conducted during May through December 1995.

The vulnerability of XM46 will play a significant role in the design of the Crusader vehicle. In some MIL-STD-2105A insensitive munitions (IM) tests, XM46 was nonreactive to bullet impact and mildly reactive to single fragment impact [1]. However, containers can react violently to shaped charge jet impact and cause neighboring containers to have similar violent reactions [2]. The violence is severe enough to destroy a vehicle. Frey et al. [2], Lyman and Blake [3], and Bates et al. [4] have reported on numerous tests to identify the mechanism leading to these reactions. In some tests, violent reactions were observed where they would not be predicted based solely on IM test results and sensitivity data. Two examples [2] are (1) single KE rod impacts at 1.60 km/s on containers with voids, and (2) double impact by fragments at 1.62 and 1.0 km/s. Based on these and other results, ARL postulated one possible ignition mechanism as shock-release-recompression [2]. Further testing supported this mechanism. It was demonstrated that judicious container design could suppress violent reactions by reducing or preventing release and recompression [3,4].

First, this report summarizes our efforts to develop a method for recovering shocked liquids. Based on these efforts, recommendations are made for future recovery testing of shocked liquids. By using the developed method, LP samples were recovered after being exposed to shocks in the ranges of 1.1–1.3, 2.4–3.6, and 5.1–6.1 GPa. The recovered samples were analyzed using Fourier transform infrared/cylindrical internal reflectance (FTIR/CIR) spectroscopy [5] and microreflectance spectroscopy. Some preliminary conclusions are drawn; however, the data are limited so more testing is recommended.

2. DEVELOPMENT OF RECOVERY TECHNIQUE

To conduct FTIR/CIR chemical analysis, approximately 3–5 ml of recovered shocked LP are required. Much of the effort described here focused on developing the recovery technique. Shock pressures as high as 7.0 GPa were desired. To develop the technique, seven tests were conducted using water as an inert surrogate for LP. Seven additional tests followed using LP. During these latter tests, additional refinement of the technique was required. In this section, we discuss all 14 tests as they pertain to the recovery technique. The chemical response of the LP is discussed in section 3.

2.1 Tests 1–7: Recovery of Water

Since shock-release-recompression is a likely ignition mechanism [2], we attempted to expose the LP to one well-defined shock wave. A schematic of our basic approach is shown in Figure 1. An explosively launched aluminum flyer plate imparted the shock. To minimize the effects of multiple shocks and rarefaction waves, the LP or water sample was held in a surround (or container) of material similar in shock impedance. Also, at least in the short term, the surround material was required to be chemically compatible with LP. Based on these criteria, polypropylene was chosen. Figure 2 shows the shock Hugoniots in the pressure-versus-particle-velocity plane for LP, water, and polypropylene. Aluminum and steel Hugoniots are shown for reference.

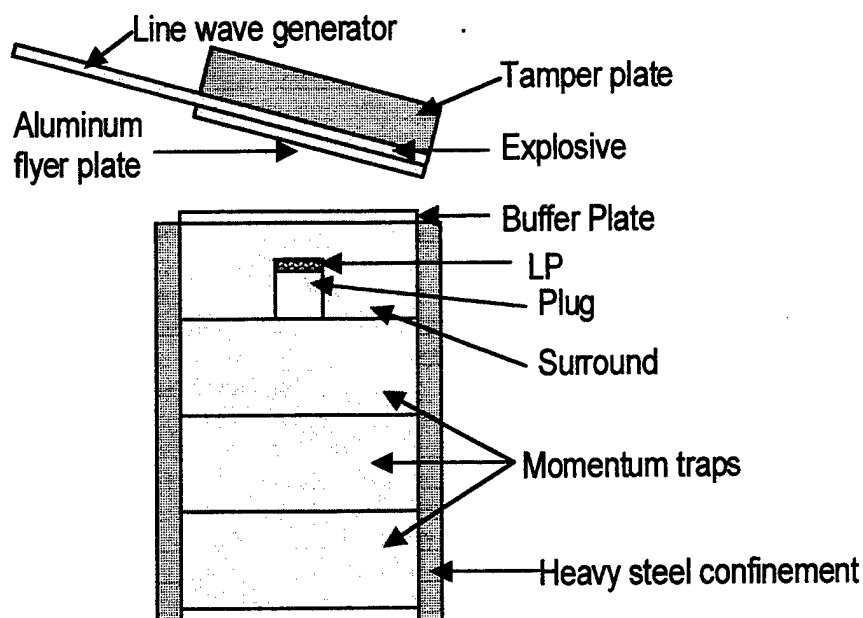


Figure 1. Schematic of general setup to recover shocked LP.

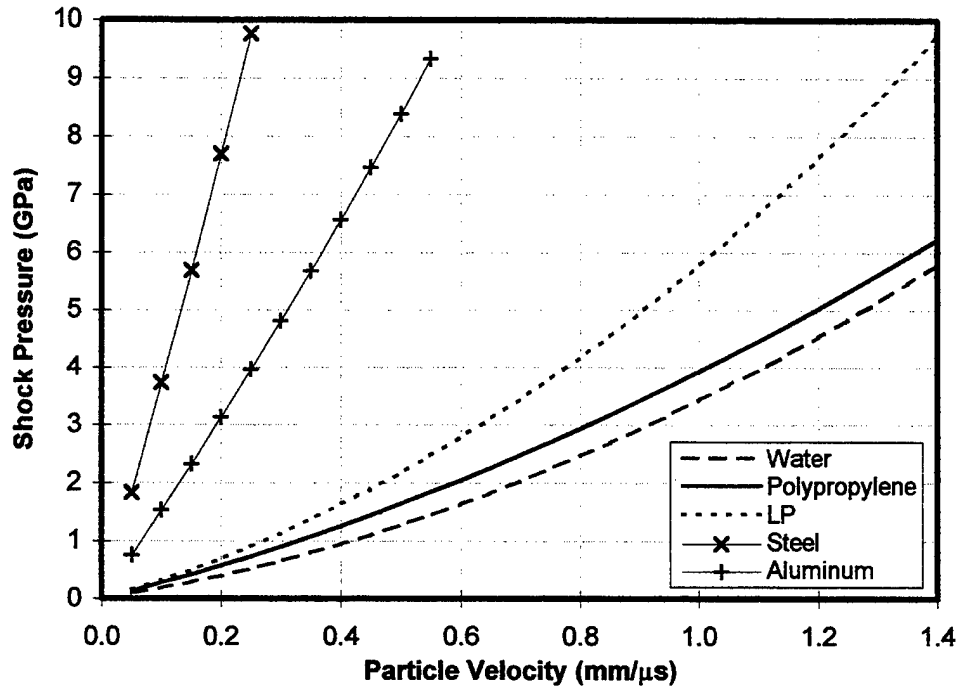


Figure 2. Shock Hugoniot curves in the pressure-versus-particle-velocity plane.

An aluminum buffer plate was used to avoid flyer-plate impact directly on the surround. The flyer-plate velocity was calculated using the Gurney equations [6] for an asymmetric sandwich. For this basic configuration, Figure 3 shows the peak shock pressure in the sample versus flyer-plate velocity. This curve was generated using the $U_s = a + bu_p$ form of the Hugoniot and impedance matching. Actual shock pressures may be lower due to the rarefaction from the trailing side of the flyer plate. Consequently, lower bounds for the shock pressure were obtained from CTH calculations. In this section, where we discuss the recovery method, the reported pressures are those calculated from the impedance match method. In section 3, where the chemical response of the LP is discussed, a range of shock pressures is reported for each test. The lower number is from CTH calculations, and the higher number from the impedance matching method.

Figure 3 shows that a velocity of ≈ 1.7 mm/μs is needed to attain 7.0 GPa in the LP. At these high impact velocities, it was difficult to generate a single shock on the sample. Heavy steel side confinement was used to diminish release waves from the lateral edges of the surround. To reduce rarefaction waves from the rear of the surround, a multiple-piece polypropylene momentum trap was placed as shown in Figure 1. Table 1 summarizes the test configurations. Several changes were usually made between tests; the major change and the qualitative recovery result are listed in the table. Each specific test is discussed in

the following sections. For each test, we only discuss the changes from the previous test. Similarly, in the schematics only the new or changed features of each test are labeled.

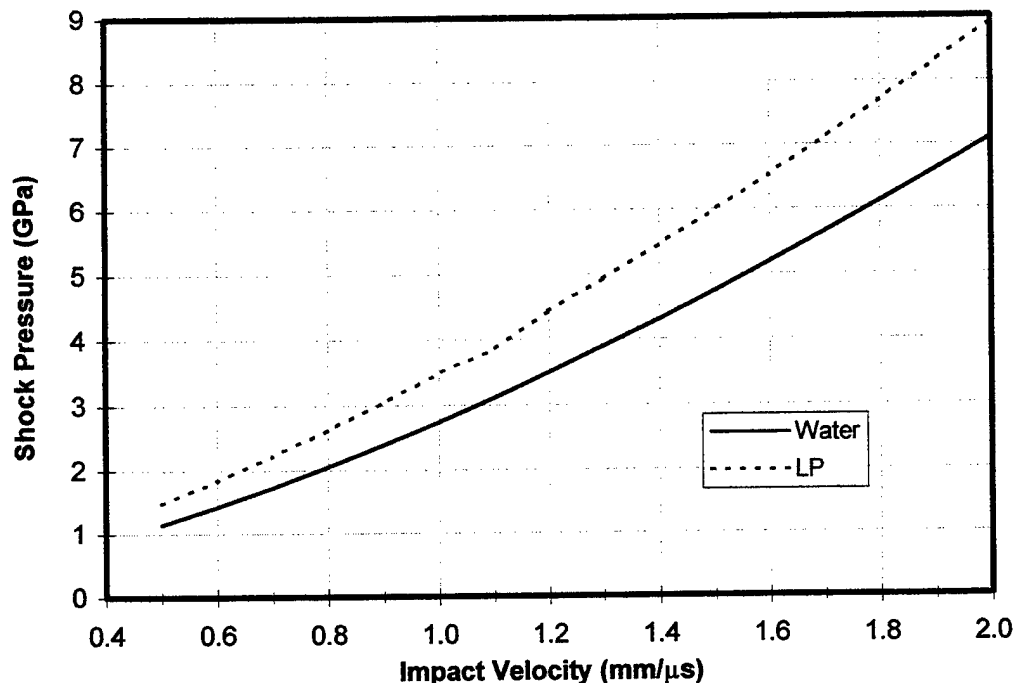


Figure 3. Shock pressure in water or LP sample as function of flyer-plate velocity for setup shown in Figure 1.

2.1.1 Test 1: Nonaxisymmetric Surround, Laterally Inserted Plug, Inertial Confinement, Buffer Dimensions Match Surround, 5.9 GPa. A sketch of Test 1 is shown in Figure 4. The flyer-plate velocity was 1.75 mm/μs corresponding to a 5.9-GPa shock in the water. A three-piece surround was used. It was 203 mm wide, 254 mm long, and 51 mm thick in the shock propagation direction. A 51-mm-long, 25.4-mm-diameter polyethylene container with a 5-ml cavity for the sample was placed in the center. An end plug was placed on both sides of the container.

The momentum trap consisted of three pieces of polypropylene also 203 mm wide, 254 mm long, and 51 mm thick. The buffer plate was 6.4-mm-thick aluminum and covered the entire surround. It was slightly recessed into the confinement. The confinement extended from the top of the surround to the center of the second momentum trap. Four pieces of 25.4-mm-thick steel were placed around the outside of the polypropylene and wrapped with duct tape in the hope that the inertial confinement would hold long enough

for passage of the shock. However, the confinement was too weak, so the plugs and container were expelled from the surround. Also, after the plugs and container blew out, the surround cracked and tore parallel to the bore.

Table 1. Major Configuration Feature or Change from Previous Test and the Qualitative Recovery Result

Test	Sample	Shock Pressure (GPa)	Major Configuration Change	Result
1	Water	5.9	Nonaxisymmetric surround; Inertial confinement; Laterally inserted plug; Buffer size matches surround	Surround cracked; Container expelled; No sample recovered
2	Water	4.8	Welded confinement; Lower shock pressure	Confinement cracked; Container expelled; No sample recovered
3	Water	4.8	Axisymmetric surround; Threaded plug	Surround pushed out back of confinement; Container plug expelled; No sample recovered
4	Water	4.8	305-mm-long two-piece confinement; Buffer diameter equals confinement diameter	Top confinement cracked; Surround recovered in bottom confinement; Deformation of plug; Some water recovered
5	Water	4.8	305-mm-long one-piece confinement; Buffer diameter equals surround diameter	Plug deformation; Explosive product gases entered cavity; No sample recovered
6	Water	4.8	Buffer diameter equals confinement diameter	Buffer plate rotated and driven into surround; No sample recovered
7	Water	4.8	Plug from rear of surround; Buffer diameter equals surround diameter	Nearly all water recovered
8	LP	6.1	No change; Test with LP	LP combustion
9	LP	2.0	Lower shock pressure; Surround not at ambient temperature	Fractured surround; No LP recovered
10	LP	2.0	Duplicate with surround at ambient temperature	Fractured surround; No LP recovered
11	LP	1.4	Lower shock pressure	Fractured surround; No LP recovered
12	LP	1.4	Redesigned LP container	Fractured surround; No LP recovered
13	LP	1.3	Enclosed entire test in steel	Recovered nearly all LP
14	LP	3.6	Higher shock pressure	Recovered small amount of LP

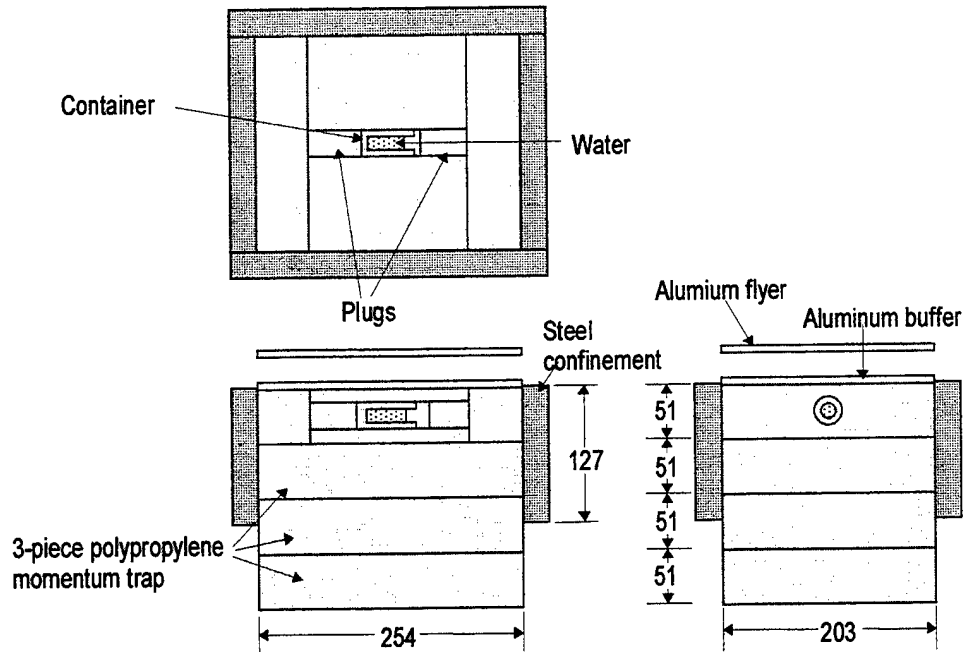


Figure 4. Schematic of Test 1. (Top, front, and right side views are shown for sections through the center of the sample. Dimensions in millimeters.)

2.1.2 *Test 2: Reduced Pressure, 4.8 GPa, Welded Confinement.* In Test 2, the flyer-plate velocity was reduced to 1.52 mm/ μ s for a shock pressure of 4.8 GPa. In an attempt to overcome the confinement problem, the steel plates were welded to angled braces. A schematic is shown in Figure 5. These changes improved the results. Although the confinement failed at one weld and allowed the plugs to escape, the container remained in the surround, and unlike in Test 1 the surround showed no evidence of splitting. Also, observed damage to the polypropylene blocks indicated that most of the momentum was removed from the surround by the momentum trap.

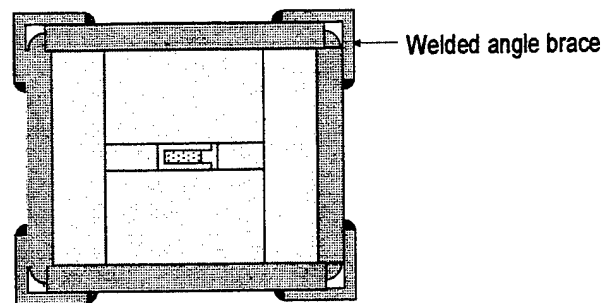


Figure 5. Schematic of Test 2 showing change in confinement from Test 1.

2.1.3 *Test 3: Axisymmetric Surround, 127-mm-Long Confinement.* At this point, confinement strength was perceived as the main problem, and the ability to keep the container in the surround was a less significant problem also to be addressed. Thus, an axisymmetric steel confinement tube approximately 19 mm thick was used for added strength. The surround diameter was 121 mm. Also, the container and plugs were threaded into the surround and to each other. Figure 6 shows a sketch of this setup.

The confinement remained intact during this test with some relatively minor plastic deformation due to radial expansion near the impact point. However, the surround came out the rear (opposite to impact) side of the confinement, and the threaded plug was expelled. Interestingly, due to the highly elastic nature of the polypropylene, there was no significant thread damage.

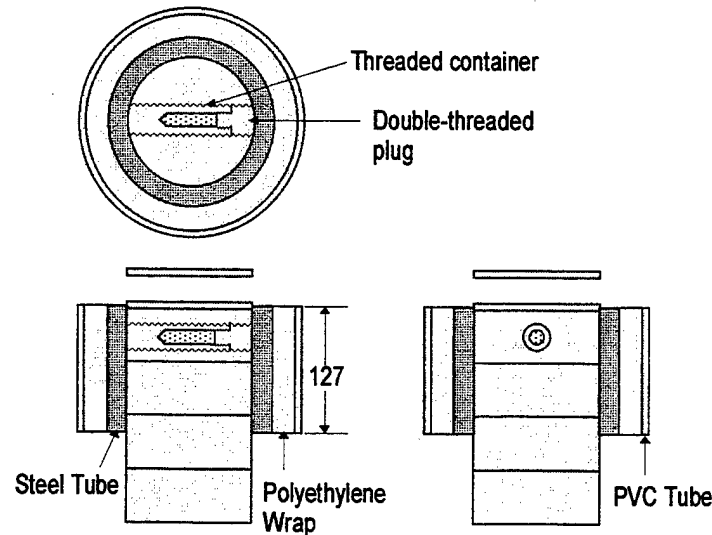


Figure 6. Schematic of Test 3. (Top, front, and right side views are shown for sections through the center of the sample. Dimensions in millimeters.)

2.1.4 *Test 4: 305-mm-Long Two-Piece Confinement, Expanded Buffer Plate.* To prevent the surround from exiting the confinement, two adjustments were made in Test 4. First, the aluminum buffer plate diameter was increased, and it was bolted to the top of the confinement. Second, an additional 7 inches of confinement were added by using a second steel tube. A sketch is shown in Figure 7. Several other minor changes were also made. The entire setup was surrounded by water to minimize lateral rarefactions, and foam was used to prevent water from hindering the performance of the momentum trap.

Following impact, the front piece of confinement fractured into four pieces due to cracks which propagated from stress concentrations at the bolt holes. The surround was pushed down into the second steel tube, which remained intact. The container and plug were recovered in the surround; however, some deformation occurred. Nonetheless, a small quantity of water was recovered from the surround.

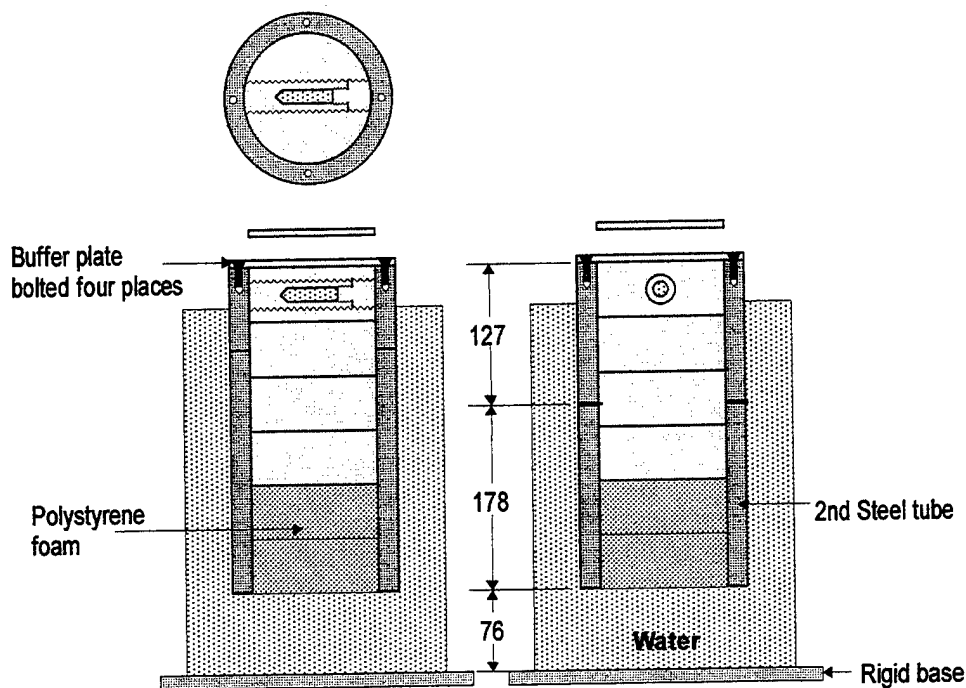


Figure 7. Schematic of Test 4. (Top, front, and right side views are shown for sections through the center of the sample. Dimensions in millimeters.)

2.1.5 Test 5: One-Piece Confinement, Buffer Diameter Equals Surround Diameter. In this test, three changes were made. First, the confinement was now a single 305-mm-long steel tube. The cover plate was reduced back to its previous configuration so its back side could be recessed slightly into the confinement. Also, the surround design was changed to one piece with a plug. A sketch is shown in Figure 8. After impact, the cover plate and surround remained in the confinement. However, some plug deformation allowed the water to escape. Also, some explosive product gases penetrated into the surround around the edge of the deformed plug.

2.1.6 Test 6: Buffer Diameter Equals Confinement Diameter. To prevent explosive gases from reaching the plug, the cover plate again was expanded out to cover the top of the confinement, but this time it was not bolted down. Also, a cement surround was used around the steel confinement in place of water

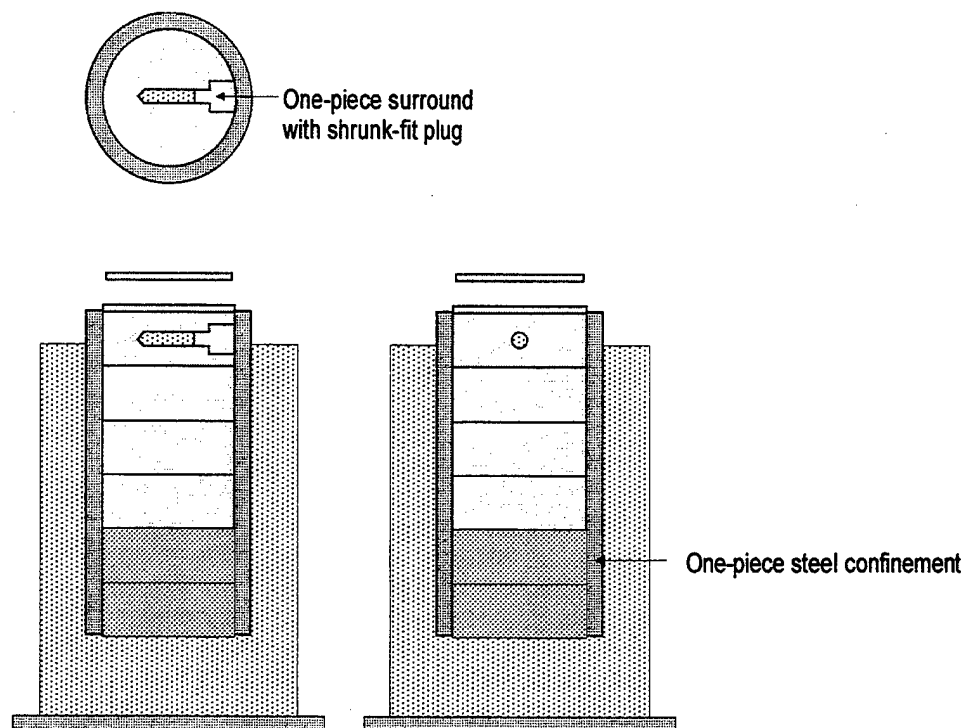


Figure 8. Schematic of Test 5. (Top, front, and right side views are shown for sections through the center of the sample.)

though this difference should not significantly affect the pressure history on the sample. Figure 9 shows a schematic. When the flyer plate impacted the cover plate, the cover did not simultaneously fracture around the entire inner diameter of the confinement. Thus, the plate rotated in the confinement and was driven into the surround after passage of the shock wave. This caused water to leak from the sample cavity.

At this point several conclusions were drawn: (1) The momentum trap successfully reduced the velocity of the surround. (2) Long confinement contained the surround during its motion. (3) The buffer plate should be the same radial size as the surround. (4) Something must be done to prevent hot explosive product gases from reaching the plug.

2.1.7 Test 7: Plug Inserted from Rear of Surround, Sample Cavity Counterbored, Buffer Diameter Equals Surround Diameter. In this test, the cover plate was moved back inside the confinement so it would not rotate and damage the surround. However, this allows some explosive product gases to enter the confinement. Therefore, a new surround design was tried with the plug inserted from the rear. A schematic is shown in Figure 10. A two-piece plug was used with a counterbored sample cavity. The

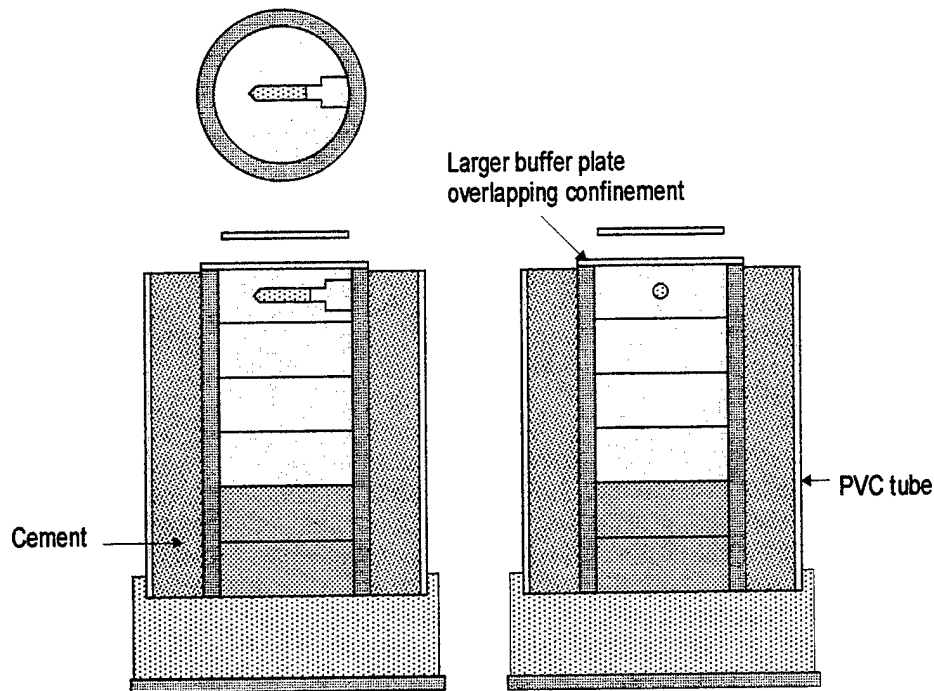


Figure 9. Schematic of Test 6. (Top, front, and right side views are shown for sections through the center of the sample.)

smaller inner plug was shrunk fit into the surround. The outer plug was threaded, and epoxy was placed on the threads. Note on the sketch that water replaced the foam under the polypropylene momentum trap. This was done in an effort to reduce the motion of the surround in the confinement. Rather than rely solely on the momentum trap to suppress motion, we now relied on the water below the polypropylene to remove some momentum.

The test was successful. The plug remained intact, and we recovered approximately 4 ml out of the initial 5-ml water sample. It appeared a successful recovery method was developed, so we proceeded to test with LP.

2.2 Tests 8–14: Recovery of XM46 Liquid Propellant

Here we discuss Tests 8–14 only as they relate to the recovery method. The chemical response of the shocked LP is discussed in section 3.

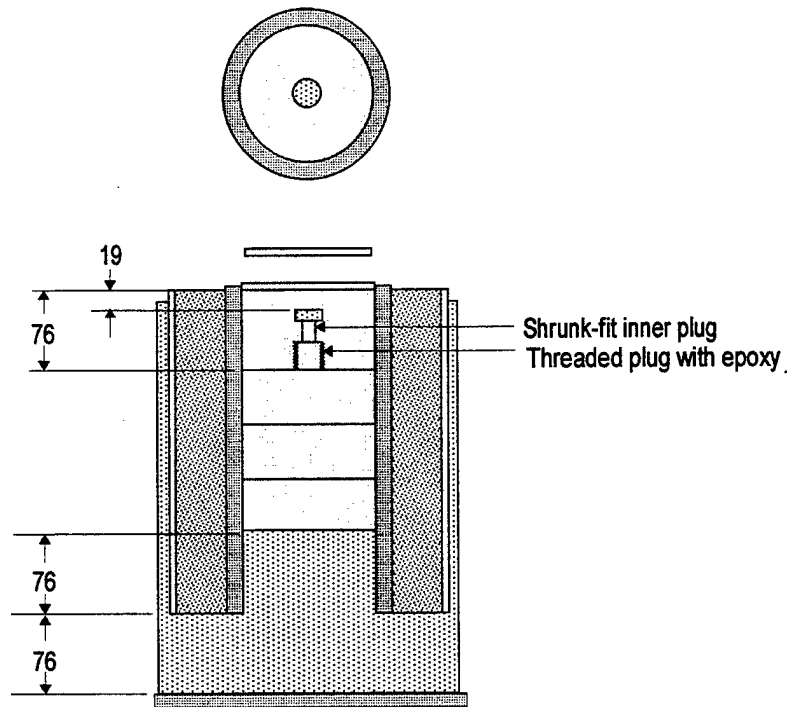


Figure 10. Schematic of Tests 7-10. (Dimensions in millimeters.)

2.2.1 *Test 8: 6.1 GPa.* Since 4 ml of water was recovered in Test 7, the setup was repeated using LP. Due to the Hugoniot difference between LP and water, the shock pressure generated by the aluminum plate impacting at $1.52 \text{ mm}/\mu\text{s}$ is 6.1 GPa in the LP as opposed to 4.8 GPa in the water. On examination of the hardware after the test, it was clear that the LP reacted vigorously. None was recovered. Since a reaction occurred at 6.1 GPa, shock pressure was lowered while keeping the same setup.

2.2.2 *Test 9: 1.8 GPa.* The impact velocity of the flyer was lowered to $0.65 \text{ mm}/\mu\text{s}$. The transmitted shock pressured was thus 1.8 GPa. The setup was the same as that shown in Figure 10. In this test, the propellant surround fractured. The fracture propagated out from the bottom corner of the LP cavity, exposing the cavity and allowing the LP to escape. No visible evidence of reaction was observed. Thus, we believe the fracture was an artifact of stresses caused solely by the shock wave. One possibility was postulated as the procedure for placing the surround in the confinement. Typically, the temperature of the surround and LP was lowered to below 0°F ; then the surround was placed in the confinement and allowed to warm and expand for a better fit. However, for this test the surround was not allowed to warm significantly prior to testing, so brittle polypropylene was suspected as a reason for fracture.

2.2.3 *Test 10: 1.8 GPa.* Test 10 was a duplicate of Test 9 except the surround was allowed to return to ambient temperature. The results were identical to the previous tests—fracture from the bottom of the LP cavity. At this point it was not clear what was causing the fracture, but clearly the cracks propagated from the stress concentrations at the corners of the cavity.

2.2.4 *Test 11: 1.3 GPa.* In an effort to eliminate fracture, two changes were made in Test 11. First, the velocity was lowered to 0.48 mm/ μ s, corresponding to a 1.3-GPa pressure. Secondly, the foam was reinserted under the momentum trap since we suspected that compression waves might be coming back up into the setup after the main shock was reflected off the base. The setup is shown in Figure 11. This test also resulted in fracture of the surround. However, now the fracture propagated from the top corners of the surround. We thus began to suspect that the impedance mismatch between the polypropylene and the LP was causing the fracture.

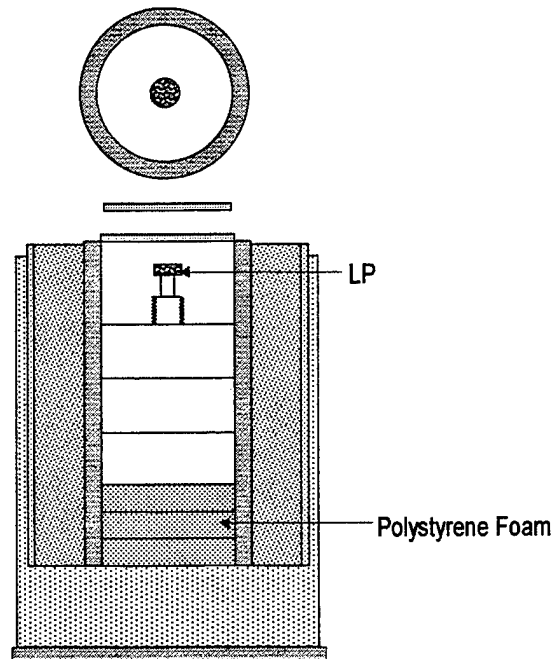


Figure 11. Schematic of Test 11.

2.2.5 *Test 12: Redesigned Surround, Sample Cavity Not Counterbored.* To reduce the stress concentration in the cavity, a new surround was designed. Rather than counterbore the cavity, the entire cavity was now made the same diameter as the inner plug. The front side of the cavity was machined

hemispherical to eliminate the stress concentrations. Also, the concrete surrounding the confinement was replaced by sand in this test. Figure 12 shows the setup. Unfortunately, this test also resulted in the surround fracturing from the top side.

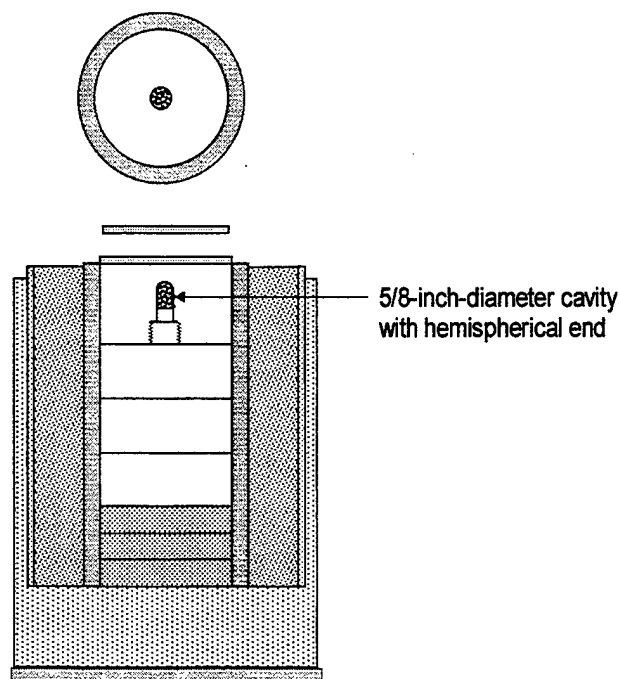


Figure 12. Schematic of Test 12.

2.2.6 Test 13: Redesigned Setup, Enclosed in Steel Case, 1.3 GPa. Due to the problem with the surround fracturing, we decided to make a radical change to the setup. The surround and momentum trap were enclosed in a steel case so that if the LP leaked out it could be recovered and perhaps analyzed, even if in a dilute solution. The setup is shown in Figure 13. The steel case was 305 mm long and had a 127-mm diameter. End plugs were machined with a 13-mm thread length, so the free volume of the container was 279 mm long. The surround design remained the same except that the diameter was reduced to 102 mm. Two pieces of 25.4-mm-thick polypropylene, 25.4 mm of water, and a third piece of 25.4-mm-thick polypropylene acted as a momentum trap. A 76-mm space was left in the bottom of the container. The container itself was surrounded by water. A stainless steel gas sampling bottle fitted with a gas sampling valve was connected to the container to capture gases in case the LP reacted violently. The sample bottle was evacuated prior to the test. A pressure gauge was placed on the hose leading to the container to detect any rise in static pressure following the test.

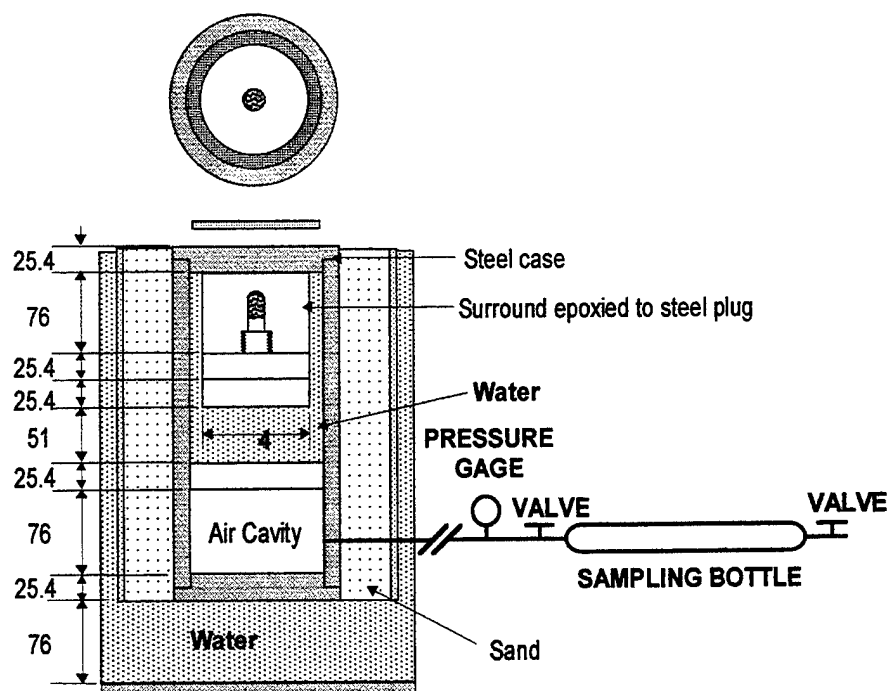


Figure 13. Schematic of Test 13. (Dimensions in millimeters.)

Since the shock pressure transmitted to the polypropylene through steel is less than through aluminum, the impact velocity was increased back to $0.65 \text{ mm}/\mu\text{s}$. The pressure on the LP was 1.3 GPa. Some initial fracture of the surround was detected in a pattern similar to the previous tests. Nonetheless, nearly all of the LP was recovered, and there was no visible evidence of reaction. The LP was analyzed using FTIR-CIR. The results are discussed in section 3.

2.2.7 Test 14: 3.6 GPa. Since LP was recovered in the previous test and no visible reaction was present, the flyer-plate velocity was increased to $1.48 \text{ mm}/\mu\text{s}$ for this test. The resulting pressure was 3.6 GPa. The gas sampling bottle was removed since the likelihood of capturing combustion gases was small.

In this test, a substantial amount of the LP was lost. Upon removal of the threaded plug, some LP residue was detected outside the inner plug. Slightly more LP was found inside the cavity, but not enough to use the FTIR-CIR technique. Thus, an infrared microscope was used to analyze the LP. The results are discussed in the following text. At this point testing ceased.

3. CHEMICAL RESPONSE OF SHOCKED LP

The tests discussed previously give results for the response of LP to five different shock levels. Ranges are given for shock pressure in most instances; the lower value is from the CTH calculations, and the higher value is from impedance matching. In only one test, at 5.1–6.1 GPa, did the LP show visible signs of reaction. Table 2 summarizes the results discussed in this section. The shock pressures were calculated assuming one-dimensional shock and a $U_s = a + bu_p$ Hugoniot for all the materials. The qualitative results and quantitative chemical analysis for these tests are discussed.

Table 2. Qualitative Results for Recovery Tests on Shocked XM46 Liquid Propellant

Test(s)	Calculated Initial Shock Pressure in LP from Impedance Matching (GPa)	Results
8	6.1	Vigorous Burn
9,10	2.0	Surround Fracture, No visible reaction
11,12	1.4	Surround Fracture, No visible reaction
13	1.3	Recovered, No visible reaction
14	3.6	Recovered, No visible reaction

For Tests 13 and 14, IR spectra were obtained with a Mattson Polaris FTIR spectrometer using Mattson/First software. For all spectra, 32 scans were collected with a resolution of 8 cm^{-1} .

3.1 Test 8: 5.1–6.1 GPa

In Test 8 the LP reacted vigorously as evidenced by the yellow-brown residue remaining after the test. Figure 14 shows a photograph of the residue inside the cavity. Note that the inner plug (recall Figure 10) is missing from the bottom cavity portion. In the top portion of the cavity, a small hole leads all the way to the impact surface of the surround. The pressure in the sample cavity during combustion must have been large enough to extrude the plug through this hole, which was no doubt larger during the combustion process when the cavity expanded. Due to the hot combustion gases, there is melted polypropylene around the hole. A bulge was formed at the impact surface of the surround from the high pressure in the cavity. No chemical analysis was performed on this test since no LP was recovered.

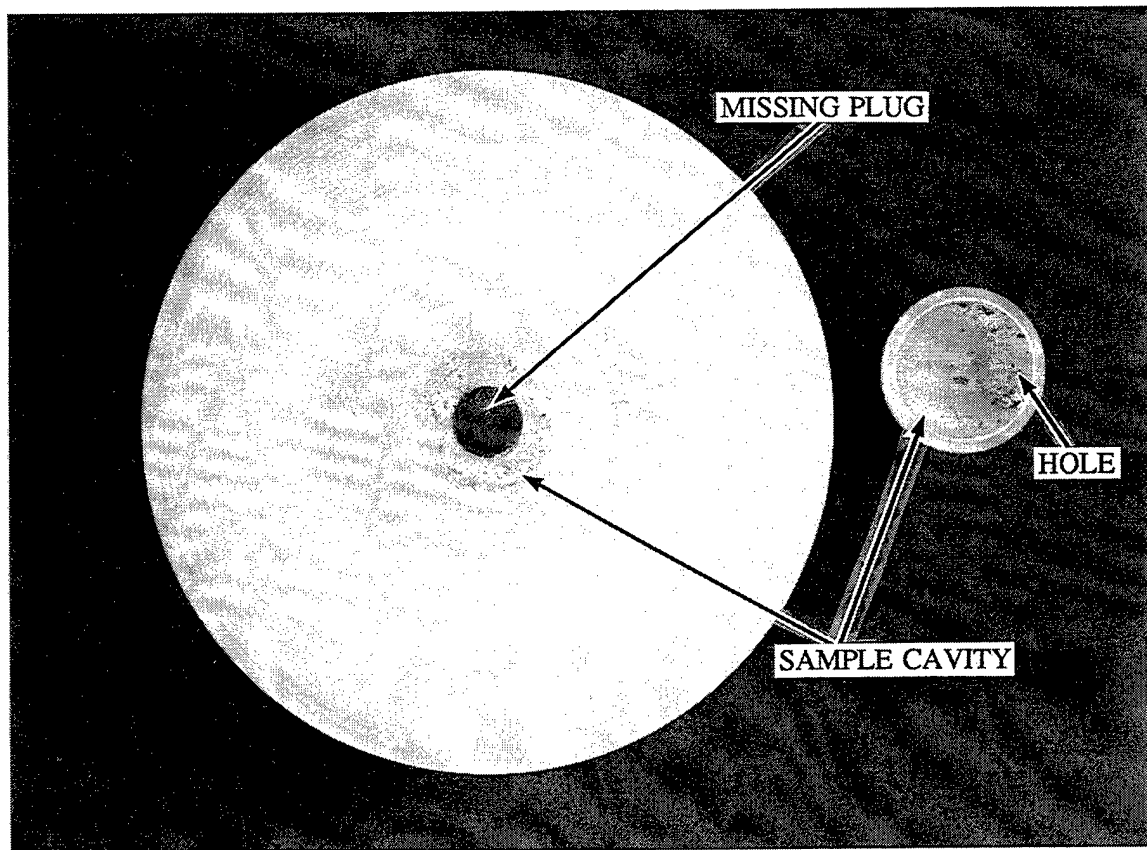


Figure 14. Photograph of LP sample cavity from Test 8. (Outside of surround has been machine to allow the cavity to be opened.)

It is interesting however that this strong reaction occurred at 5.1–6.1 GPa, particularly since the wedge test data of Idar et al. [7] show that LP is shock insensitive up to 14 GPa (detonation did not occur in an 88-mm run distance). This illustrates why we are reluctant to draw firm conclusions based on these limited data. Several possible explanations exist for the combustion.

- (1) A hot fragment entered the top of the surround and created the observed hole. We doubt this scenario since it would be difficult for a fragment to find its way under the center of the buffer plate and have any significant velocity. Also, if a fragment did perforate the surround, evidence of impact would be seen on the back of the cavity since the LP offers little resistance to the fragment.
- (2) The water did not disperse the shock sufficiently, so a compression wave reflected from the rigid base and propagated back up the polyethylene pieces to ignite the propellant,

which was sensitized by the initial shock wave. This is analogous to larger scale vulnerability tests.

- (3) The shock pressure alone is sufficient to cause initiation, and the confinement then allowed the reaction to build. Though LP is insensitive to single shocks at pressures higher than 6.1 GPa, in those previous tests the sample was unconfined. In the current setup, even a small amount of gasification by the propellant will cause significant pressurization and a large increase in burning rate.
- (4) An air bubble in the cavity sensitized the propellant. Using the counterbored setup (recall Figure 11), it is difficult to be sure that no bubbles exist in the sample. Since bubbles of the size possible in this test are regularly present in large vulnerability tests where explosions do not occur, we doubt that bubbles lead directly to ignition. It is possible however that a small amount of reaction in the vicinity of a bubble could start a process of pressurization, leading to ignition as in the third scenario. Thus, the confinement may have lowered the critical bubble size.

3.2 Tests 9–12: 1.1–2.0 GPa, No Recovery

Tests 9 through 12 can all be placed into one category—no significant reaction but no LP recovery. In each of these cases, the surround fractured and allowed the LP to escape. The fractures always started from a point of stress concentration, such as a sharp corner, if it existed. No visible evidence of burning was seen.

Two possibilities exist for this behavior. One is that outgassing of the LP creates pressure with little temperature rise, thus allowing the surround to fracture with no combustion. The second is that fracture occurs due to high stresses caused by the small impedance mismatch between the LP and the polypropylene. We believe the latter explanation is more probable since Tests 13 and 14 somewhat contradict the former.

3.3 Test 13: 1.1–1.3 GPa

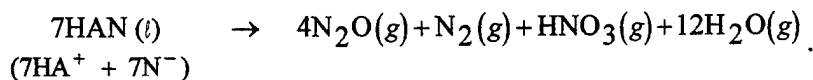
As with Tests 9–12, no visible evidence of reaction was seen in Test 13. Some initial fracture was seen in the surround in a similar pattern to Test 12. In this case nearly all of the LP was recovered. This

supports our position that stresses from the impedance mismatch are responsible for fracture, since if fracture was due to pressurization by outgassing it would be independent of the confinement change from Test 12 to 13.

The gases sampled from the enclosure (recall Figure 13) were identified with a Hewlett-Packard GC-MS system (Model 5890 GC, Model 5970 MSD). A gas-tight syringe (5- μ l volume) was used to deliver gas samples to the GC injection port. The analysis showed that the gases were air, which is not unexpected since nearly all of the LP was recovered.

The recovered LP was analyzed using FTIR-CIR spectroscopy. A sample of unshocked LP was also analyzed. These "Circle-Cell" measurements were performed using a Barnes CIR accessory with an 8-cm-long by 6.3-mm-diameter zinc selenide crystal rod sealed to a standard glass cell with Teflon O-rings. The cell was filled with 3 ml of sample via a disposable glass pipette inserted through the filling port until it almost touched the rod. Care was taken to exclude air bubbles from the sample chamber.

Spectra from the unshocked and shocked LP are compared in Figure 15. The assignments for the peaks are shown on the figure. Note that there are some small differences in the nitrate (labeled N⁻) peak between 1300 and 1400 cm⁻¹. These small differences are attributed to background variations caused by IR absorption by atmospheric moisture. Such differences have also been observed in the spectra of unshocked LP. Had there been any substantial reaction, a decrease in the relative intensity of the N⁻ peaks compared to other peaks would occur due to decomposition of nitrate ions. Similarly, decreases in the HA⁺ and TEA⁺ would be observed. For HAN, the following decomposition reaction might be expected [8]:



The appearance of new peaks between 1700 and 1800 cm⁻¹ would have indicated oxidation of the organic component of the LP (i.e., triethanolamine, (HO-CH₂-CH₂)₃-NH⁺, which is expressed as TEA⁺ in Figures 15 and 16).

Based on the current understanding of LP thermal reactions, it is expected that HAN would decompose before TEAN. In an IR spectrum, this would be reflected by an increase in TEA⁺ peaks relative to HA⁺ peaks. Such an increase is not observed in Figure 15. This suggests that the LP did not decompose.

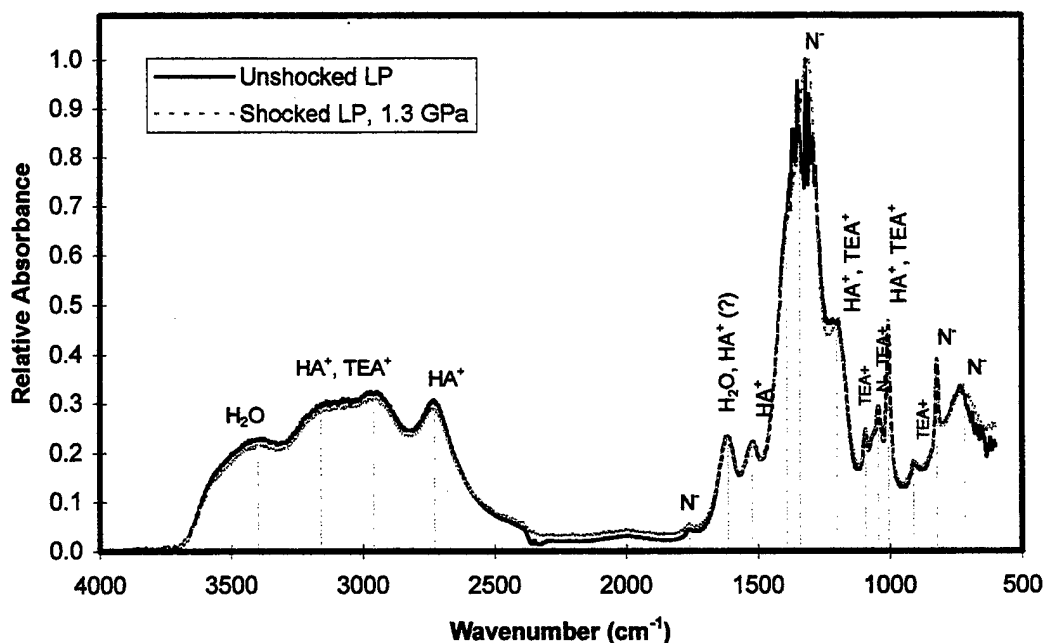


Figure 15. Spectra from unshocked LP and LP shocked to 1.1–1.3 GPa in Test 13.

3.4 Test 14: 2.4–3.6 GPa Shock

The detection of the onset of fracture in Test 13 did not give us much confidence in elevating the shock pressure. Nonetheless, Test 14 was conducted at a 2.4-GPa to 3.6-GPa shock level. A similar fracture pattern was found in the surround, but it remained intact, and a small quantity of LP was recovered. Some LP was recovered from the cavity, and some additional LP was recovered from the region between the inner plug and the threaded plug. These are referred to as “clean” and “dirty” shocked LP, respectively. The recovered amounts were too small for FTIR-CIR, so microreflectance FTIR spectroscopy was used instead.

The microreflectance spectra were obtained using a Spectra-Tech IR-Plan IR microscope accessory with a mercury-cadmium-telluride (MCT) detector. The microscope was operated in reflectance mode using aluminum foil as a background. Kramers-Kronig transformations [9] were used to correct for distortions caused by specular reflectance. Material was removed from the shock-impact sample cavity by means of a disposable glass pipette. Recovered LP was placed directly on a piece of aluminum foil and immediately analyzed. It must be noted that since this technique is not the best one for analysis of liquid

samples, one must expect both baseline variations and loss of information in the 1800–4000 cm^{-1} wavelength range.

Figure 16 shows the spectra for unshocked, "clean" shocked, and "dirty" shocked LP. Analysis of the "clean" drop resulted in an infrared spectrum that was very similar to that of unshocked LP. The clean and unshocked spectra were normalized using the N^- peak at $\approx 1340 \text{ cm}^{-1}$. While baseline variations seem to indicate a change in chemical composition, comparison of HA^+ peaks near 1500 cm^{-1} and 1000 cm^{-1} , of TEA^+ peaks at 1000 cm^{-1} , and of N^- peaks at 825 cm^{-1} and $1300 - 1400 \text{ cm}^{-1}$, suggests no significant change in composition. Thus, it is concluded that the "clean" LP experienced no decomposition and that variations in the spectra are the result of spectral distortion caused by the sampling technique.

Analysis of the "dirty" drop, however, yielded an infrared spectrum which was quite different from that of the clean drop. When normalized to the HA^+ peak near 1500 cm^{-1} , or the HA^+ and TEA^+ near at 1000 cm^{-1} , it appears that there is a significant decrease in the N^- absorbances between 1300 and 1400 cm^{-1} . However, the N^- absorbances at 825 cm^{-1} appear to be approximately equal. Based on these inconsistent results, it is difficult to draw a conclusion, except perhaps that additional data should be collected under conditions with a 3.6-GPa shock.

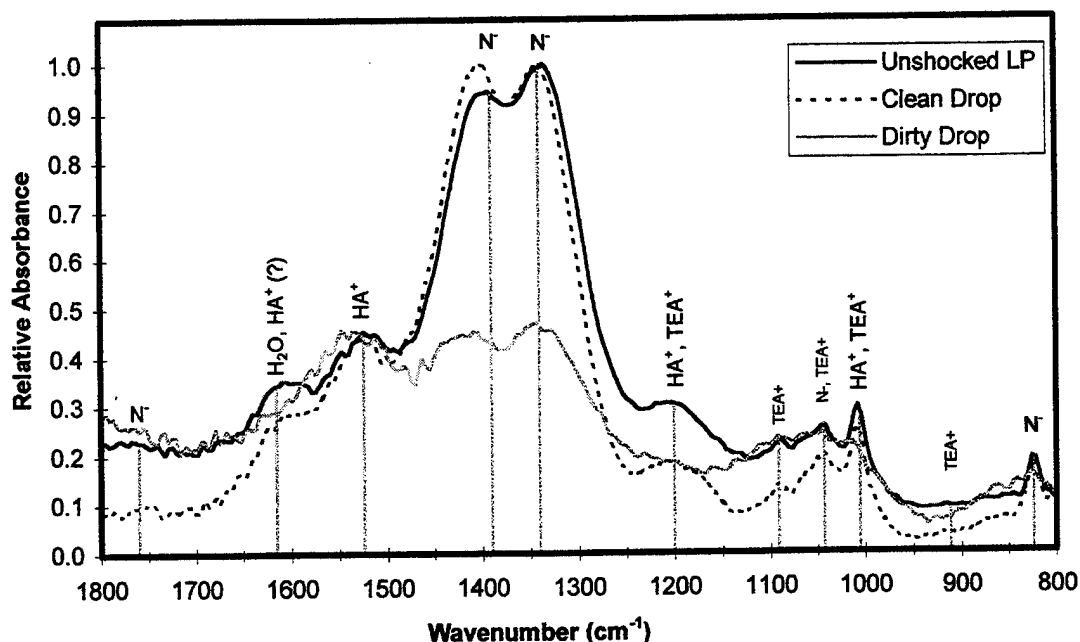


Figure 16. Spectra from unshocked LP and LP shocked to 2.4–3.6 GPa and recovered from both inside the sample cavity ("clean") and outside of the sample cavity ("dirty"). (The clean drop was normalized to unshocked LP using the N^- peak at $\approx 1340 \text{ cm}^{-1}$. The dirty drop was normalized to unshocked LP using the HA^+ peak at $\approx 1524 \text{ cm}^{-1}$.)

4. CONCLUSIONS AND RECOMMENDATIONS

Based on the results presented here, several conclusions can be drawn about techniques for recovering shocked liquid samples and the single shock response of XM46 liquid propellant.

In order to recover shocked liquids, we recommend a setup containing the following features:

- (1) The flyer plate should be large enough to prevent edge effects from influencing the sample prior to passage of the entire shock wave. Consequently, the sample should be close to the impact surface of the flyer plate.
- (2) A momentum trap should be used to prevent rarefactions from traveling back into the surround and influencing the results of the test.
- (3) Strong confinement around the outside of the surround is required to diminish lateral release waves. This confinement should be long enough to contain the surround during any motion that may occur. By using a hydrocode, a test setup could be engineered to minimize the effects of any wave reflections.
- (4) An impedance-matched surround should be used to hold the liquid specimen.

Based on our observations, a very close impedance match is required. It is quite possible that even the small impedance mismatch between LP and polypropylene caused fracture where it did not occur for similar tests with water, which has a Hugoniot closer to that of polypropylene (recall Figure 2). For example, the 5.06-GPa shock (computed by impedance matching) in the polypropylene surround for Tests 2-8 will drop to a 4.83-GPa shock in the water but rise to a 6.09-GPa shock in LP. The latter sets up a 1.03-GPa shock differential between the side of the sample and surround, whereas the former leads to only a -0.23-GPa difference. This behavior may explain the cracking of the surround in Tests 9-14. The absence of cracking in Test 8 is still a question, but perhaps the combustion somehow influenced the wave interactions and stresses in the surround. One possible method to obtain a closer impedance match may be mixing water and LP to create a solution of desired shock impedance.

As far as the chemical response of the LP to a single shock is concerned, these data indicate that there is no significant reaction to a single shock up to 2.4-3.6 GPa, and at 5.1-6.1 GPa a combustion resulted.

However, as stated previously, these conclusions must be considered within the context of the limited data and the ongoing development of the test method. It is certainly possible that the combustion at 5.1–6.1 GPa did not result from the incident shock but instead from some other artifact of the test. The nonreaction at less than 1.1–1.3 GPa we believe to be a firm conclusion since FTIR-CIR generated nearly identical spectra for shocked and unshocked LP. The microreflectance spectra obtained from the sample shocked to 2.4–3.6 GPa indicate no reaction occurred. However, our confidence in this result is not as high since the integrity of the sample was compromised during the test. Indeed, the LP that was recovered outside the sample cavity gave some inconclusive results based on its spectra. Thus, more testing is required to definitively determine whether chemical reaction occurs during the passage of a single shock wave through virgin LP.

However, based on these data, we conclude, albeit with some reservations, that no sensitizing chemical reactions occur in the initial shock phase of the shock-release-recompression mechanism if the shock level is less than 2.4 GPa. Consequently, in vulnerability tests where the incident shock is less than 2.4 GPa but a violent reaction results, sensitization is solely a mechanical phenomenon occurring during release, and initiation occurs by recompression of otherwise previously unreacted LP.

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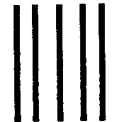
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